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PATENT SPECIFICATION

1,093,544



NO DRAWINGS

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Inventor: CHARLES DONALD FORREST

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Int. Cl.:—C 06 b 15/02

COMPLETE SPECIFICATION

Explosive

We, E. I. DU PONT DE NEMOURS AND COMPANY, a corporation organised and existing under the laws of the State of Delaware, United States of America, of Wilmington, 5 State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the 10 following statement:—

This invention concerns improvements in and relating to detonating explosive compositions.

British Patent Specification No. 857,175 15 describes flexible detonating explosive compositions consisting essentially of PETN (pentaerythritol tetranitrate), of specified very fine particle size and in amount at least 44%, soluble NC (nitrocellulose), of average DP (degree of polymerization) 2000 to 3000 and in amount 6.5 to 14%, and plasticiser such as a trialkyl (C₂ to C₈) ester of 2-acetoxy-1,2,3-propanetricarboxylic acid, in amount 15 to 20 35%, such as have excellent explosive properties and can readily be formed into sheets. Nevertheless, we have found that similar explosive compositions based on RDX and/or HMX according to the present invention have 25 similarly good explosive and physical properties, and are even superior in certain respects, especially at higher explosive loadings.

According to the present invention, there is provided a detonating explosive composition comprising a substantially uniform blend consisting essentially of crystalline cyclotrimethylenetrinitramine and/or cyclotetramethylenetrinitramine, in amount up to substantially 85%, and preferably at least 65, especially 68, to 80%, and of average particle 35 size below 200 microns, high viscosity soluble nitrocellulose in amount substantially ½ to 40 15%, preferably 2 to 8%, and a plasticizer for nitrocellulose being a phosphate ester of

general formula O=P—(OR)₃ and/or a carboxylate ester of general formula 45

X(OOCCH₃)_m(OOCR)_k,

wherein the R groups may be the same or different and are C₁ to C₈, preferably at least C₄, functionally-unsubstituted (i.e. inert in the system in the sense that the mono-hydroxy compounds ROH have no reactive groups other than the OH groups) aliphatically-saturated organic, preferably hydrocarbyl, radicals, X is a C₁ to C₆ hydrocarbon nucleus, preferably saturated, particularly saturated acyclic, or a benzenoid nucleus, to which the ester groups are attached, m is either 0 or 1 and k is either 2 or 3, in amount substantially 10 to 34.5%, preferably 12 to 30%, all percentages being by weight.

Cyclotrimethylenetrinitramine is commonly referred to as RDX, hexogen or cyclonite. Cyclotetramethylenetrinitramine is commonly referred to as HMX, octogen or homocyclonite. Mixtures of these nitramines may be used, e.g. the reaction products formed by subjecting hexamethylenetetramine, or the precursors, formaldehyde and ammonium nitrate, to nitrolysis. As the explosive loading drops, the compositions tend to be harder to initiate, to have lower explosive strengths and detonation velocities and eventually even fail to propagate detonation reliably. As the explosive loading increases the compositions tend to become more difficult to mix and shape and to have less desirable physical properties. The average particle size, referred to herein, means weight average particle size, as calculated from standard sieve analysis values and from microscopic measurement values for particles generally smaller than the standard 325-mesh sieve apertures of 43—44 microns. In general, the initiation sensitiveness of nitramines of average particle size below 100 microns is

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[Price 4s. 6d.]

better, and nitramine crystals of such relatively smaller particle sizes are generally preferred, especially up to 75 microns although generally, they will be at least 1 micron in diameter.

The high viscosity soluble nitrocellulose is described in British Patent Specification No. 857175 and should generally be of average DP substantially 2000 to 3000 and of nitrogen content 7, preferably 9, to 13%. By soluble is meant solubility in a mixture of ethyl ether and ethyl alcohol.

Suitable phosphate and carboxylate esters include triethyl phosphate, tris(2 - ethylhexyl) phosphate, tris(tetrahydrofurfuryl) phosphate, diisooctyl adipate, bis(2-ethylbutyl) azelate, bis-(2-ethylhexyl) phthalate, triethyl 2-acetoxy-1,2,3-propanetricarboxylate, tris(2-ethylhexyl) 2 - acetoxy - 1,2,3 - propanetricarboxylate, and preferably tributyl phosphate, dibutyl phthalate, bis(2-ethylhexyl) adipate and bis(2-ethylhexyl) sebacate, and especially tributyl 2-acetoxy - 1,2,3 - propanetricarboxylate and other alkyl esters of 1,2,3 - propane tri-carboxylic acid. More than one ester may, of course, be present, and thus, the ester may consist at least in part of one or more of these esters and optionally one or more other esters.

In selecting any particular plasticizer or plasticizer mixture for any particular composition of the invention, the plasticizer should desirably have a negligible rate of evaporation in the composition, be liquid over the temperature range of use, e.g. -40 to +160°F., have a low solubility in and solvent power for water, and be substantially a non-solvent for the nitramine selected, but be at least partially miscible, and preferably completely miscible, with the grade of nitrocellulose employed, and should maintain relatively constant properties with time and over the temperature range to which it is to be exposed. Further details are given in Chapter 15 of *The Technology of Solvents and Plasticizers* by A. K. Doolittle, New York, John Wiley and Sons, Inc. (1954). When the nitrocellulose concentration is about 2 to 8%, especially 2 to 4% of the total explosive composition, it is preferred to use at least 4:1 ester:NC to get good flexibility at low temperatures, including subzero temperatures.

The explosive compositions of the present invention may be prepared as described in British Patent Specification No. 875175, but it is preferred first to form a uniform blend of some, e.g. 50 to 90%, especially 65 to 85%, by weight of the nitramine and substantially all of the plasticizer, and then to blend in the NC until no lumps or inhomogeneities (uncolloided NC) are present before adding the rest of the nitramine and blending to get uniformity, and preferably to make small unconsolidated granules. Elevated temperatures, such as 100 to 150°F. (38 to 65°C.), preferably 120 to 140°F. (50 to 60°C.), generally facilitate blending and it is therefore preferred that the blending should be carried out at least in part at an elevated temperature. The nitramine may first be conventionally wetted with water or another non-solvent, to reduce hazards, but at least some, e.g. at least one-sixth, or all of the nitramine is preferably pre-coated with the NC plasticizer, e.g. in amount 5 to 25% of the weight of nitramine/ester combination, especially if moderately fine or very fine particles are used. The use of the NC plasticizer avoids introducing extraneous ingredients into the explosive composition. Drop test values (measured as minimum distances a 5kg weight can drop onto the test samples in a modification of the apparatus described in U.S. Bureau of Mines Bulletin 346 (1931) and cause detonation in 10 out of 20 samples) of very fine, below 44 microns, RDX and HMX are increased from 12—14 and 6—8, respectively, without any ester coating, to 54 and 43, respectively with 5% coatings and to a maximum of over 56 and 53, respectively, with 10% coatings of the tributyl 2 - acetoxy - 1,2,3 - propanetricarboxylate, which means that the impact sensitivities are correspondingly reduced by these ester coatings.

The NC may also be used wetted with water and/or alcohol, but, provided that careful precautions are taken to avoid the resultant increased hazard, it may be preferred to use substantially dry NC, especially when no water or other extraneous ingredient is used with the nitramine.

The resulting generally granular explosive products may readily be converted into compact, self-supporting yet flexible articles such as sheets, including tapes, cords, blocks and other rectilinear and curvilinear shaped articles, including those of relatively complex configuration, e.g. having V-grooves or cavities, by conventional processes including pressure molding, rolling and extrusion, at ambient or elevated temperatures, depending on the particular composition and type of forming involved, and may be cut or sliced to give special configurations not obtained by the usual forming procedures. Sheets of 0.05 to 0.5 inch (1.25 mm to 1.25 cm) in thickness at loadings of 1.3 to 14 grams per square inch of surface area, and cords of diameter 0.125 to 0.75 inch ($\frac{1}{3}$ cm to 2 cm) at loadings of 0.3 to 1.2 grams per inch of length are especially suitable.

The following Examples further illustrate the invention; all parts and percentages are by weight.

EXAMPLE 1

RDX is premixed with an agitator in a water-jacketed mixer at about 130°F. (54°C.) with 15 to 20% of water, and 15 to 25% of the total is removed from the mixer. The heated wet nitramine remaining in the mixer is blended for several minutes with the ester

(tributyl 2-acetoxy-1,2,3-propanetricarboxylate), in the proportions indicated in Table 1, until a uniform mixture is obtained. Free water in the mixture is then decanted, and a small amount (0.2—0.5% of the total composition) of an inert pigment or dye, e.g. lead chromate, is added, if desired, to impart a distinctive color to the final composition. Nitrocellulose, in the proportions indicated in Table 1, having a nitrogen content of about 12.3% and a degree of polymerization around 3000 is then incorporated in three portions at intervals of a few minutes, the portions constituting about $\frac{1}{2}$, $\frac{1}{4}$ and $\frac{1}{4}$ of the total nitrocellulose, respectively. The heating and agitation are maintained during and after this addition until all lumps or inhomogeneities are gone, signifying the absence of uncolloided nitrocellulose, and until the mixture is substantially water-free when the remaining 15 to 25% of the wet crystalline cyclotrimethylenetrinitramine is incorporated, and the mixture is heated and agitated for several additional minutes, until the mass is converted into small, substantially dry, non-consolidated granules, which are removed from the mixer and passed to an extruder to form tough yet flexible sheet explosive products characterized by a high degree of uniformity in thickness, composition, and consistency. The explosive characteristics are assessed for sheet products of the same thickness, 0.16 inches (4 mm), except for composition 6 which is of thickness 0.32 inches (8 mm) and at equivalent weight loadings per unit of area, by measuring the velocity of detonation of the sheets when initiated by the indicated initiators.

Compositions 1 to 4, inclusive, contain very fine crystals (average particle size, 7—10 microns) of RDX, whereas composition 5 contains crystals of somewhat larger size (average particle size, 70—80 microns), and composition 6 contains relatively coarse crystals (average particle size, 170—180 microns).

Composition No.	1	2	3	4	5	6
% RDX	68	70	72	75	70	70
% Ester	26	25	24	20	25	25
% NC	6	5	4	5	5	5
Density, g/cc	1.52	1.52	1.53	1.58	1.51	1.53

Detonation Test

Initiator	(a)	(a)	(b)	(a)	(a)	(c)
Velocity of Detonation, meters/sec	7250	7460	7250	7460	7170	6570

(a) Blasting cap with base charge of 12 grains of PETN.
 (b) Blasting cap with base charge of 9 grains of PETN.
 (c) 8-Gram pellet of waxed cyclotrimethylenetrinitramine.

Sheet explosive products having similar explosive and physical characteristics are obtained when tributyl phosphate, dibutyl phthalate, bis(2-ethylhexyl) adipate, or bis(2-ethylhexyl) sebacate is substituted on an equivalent weight basis for the tributyl 2-acetoxy-1,2,3-propanetricarboxylate in the above compositions. These formulas also can be shaped into tough yet resilient and flexible sheet products by cold or hot rolling procedures, or alternatively, they can be converted into tapes, cords, and similar configurations, for example, by extrusion through suitable dies.

EXAMPLE 2

Following the general procedure of Example 1, a composition (Composition No. 7) of 70% cyclotetramethylenetrinitramine having an average particle size in the 12—15 micron range, 22% of tributyl 2-acetoxy-1,2,3-propanetricarboxylate, and 8% of the high-viscosity, soluble nitrocellulose of the compositions of Example 1 is prepared by first adding all the tributyl ester to the fine-particle cyclotetramethylenetrinitramine and allowing the mixture to stand for a period sufficient for the ester to coat and soak into

the nitramine crystals. At a temperature of approximately 140°F. (60°C.), the nitrocellulose, in water-wet state, then is incorporated into the coated crystals in approximately three equal portions. Mixing is continuous at the specified temperature until the nitrocellulose is thoroughly blended in and the mixture is substantially dry. The mass then is removed from the mixer and extruded through dies to form explosive cords of various diameters. One cord product so obtained has a diameter of 0.46 inch (12.5 mm) and a density of 1.58 g/cc. When initiated with the aid of an 18-gram pellet of waxed cyclotetramethylenetrinitramine, a 44-inch (1.1 m) length of this cord detonates completely at a velocity of 7350 meters per second.

Finished cyclotetramethylenetrinitramine-containing explosive products of substantially the same physical and explosive properties are obtained when the foregoing procedure for preparing the subject formula is modified by withholding from the blending process a portion of the ester-coated cyclotetramethylenetrinitramine, e.g., about from 10 to 25% thereof, until after the nitrocellulose is added and thoroughly blended in. The more granular product so obtained is, however, somewhat easier to handle and fabricate into void-free shaped articles such as cords, tapes and sheets.

EXAMPLE 3

Following the general procedure of the foregoing Examples, the compositions of follow-

ing Table 2 are prepared by thoroughly blending and coating the specified amount of cyclotetramethylenetrinitramine (HMX) in Table 2, and having substantially all particles smaller than 50 microns and an average particle size of less than 8 microns) with the specified proportion of tributyl 2 - acetoxy - 1,2,3 - propanetricarboxylate ("ester" in Table 2) and setting aside a portion of approximately 30% of the resulting mixture for later use. Then, at a temperature of about 140°F. (60°C.), the total quantity of nitrocellulose (NC) specified is added in dry form in several portions to that part of the cyclotetramethylenetrinitramine-ester mixture remaining in the mixer, and mixing is continued at the same temperature until the mass is substantially free of uncolloided nitrocellulose. The portion of the cyclotetramethylenetrinitramine - ester mixture previously reserved is incorporated into the mixture, and the resulting mass is agitated until small, non-consolidated granules are obtained. During the last few minutes of mixing, the mixer is maintained under vacuum to allow entrapped air to escape from the mass. The product then passes to an extruder that is fitted with dies of various sizes to prepare cord products ranging from 0.125 to 0.25 inch (approximately 3 to 6 mm) in diameter, whose densities are determined at the same diameter and at equivalent weight loadings per unit of length and whose detonation velocities are measured when initiated by a 10-gram PETN primer.

TABLE 2

Composition No.	8	9	10
% HMX	80.0	85.0	85.0
% Ester	17.5	13.5	14.5
% NC	2.5	1.5	0.5
Density, g/cc	1.64	1.67	1.69
 Velocity of Detonation, meters/sec			
0.125-in. (3 mm) cord	7350	7620	8020
0.187-in. (4.5 mm) cord	7640	7875	8100
0.250-in. (6 mm) cord	7975	8195	8250

EXAMPLE 4

70 Table 3 contrasts explosive and physical characteristics of several explosive compositions of this invention with characteristics of explosive compositions of the type disclosed in British Patent Specification No. 857175.
 75 The crystalline high explosives are of comparable very fine particle size, i.e. substantially

all of the particles are smaller than 44—50 microns. The average particle size of the PETN is about 7 to 10 microns, and that of the RDX and HMX is 8 microns or less. The "ester" in the compositions of Table 3 is tributyl 2 - acetoxy - 1,2,3 - propanetricarboxylate.

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TABLE 3

Compositions of This Invention	Density, g/cc	VOD ^(a) .meters/sec	Ambient Temp	Flexibility ^(b) at Low Temp
11. 63/29/8% RDX/Ester/NC	1.48	7150	Good	Good, at -40°F & -65°F
12. 68/26/6% HMX/Ester/NC	1.52	7250	Good	Good, at -40°F & -65°F
13. 70/22/8% HMX/Ester/NC	1.58	7350	Good	Good, at -37°F
14. 72/23/5% RDX/Ester/NC	1.55	7460	Good	Good, at -40°F & -65°F
15. 80/17.5/2.5% HMX/Ester/NC	1.64	7460	Good	Good, at -40°F
Compositions of British Patent Specification No. 857,175 (for Comparison)				
X 67/27/6% PETN/Ester /NC	1.47	6900—7000	Fair	Poor, at -20°F
Y 71/24/5% PETN/Ester/NC	1.48	7000	Poor	Poor, at -10°F
Z 80/17.5/2.5% PETN/Ester/NC	1.53	(c)	Poor	Poor, at -40°F

(a) Velocity of detonation

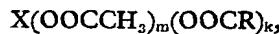
(b) Flexibility at ambient temperature (about 70—75°F) is determined by manual twisting and bending of the sheet or cord product in the as-made state. Low-temperature flexibilities are determined after at least several hours' storage of the sheet or cord at the indicated subzero temperature or temperatures. The low-temperature test results reported are based on a quick folding of the chilled specimen around a 1/2-inch wood dowel so that it is bent back on itself essentially 180°, followed by immediate visual inspection of the sample for crazing, cracks and similar flaws. In the Table, "good" indicates absence of brittleness and flaws; "fair" means moderate brittleness; and "poor" indicates significant brittleness and cracking-crazing flaws.

(c) Cord product is too fragile to permit detonation velocity determinations on 10—15 inch lengths generally used for this test.

Table 3 shows that these compositions of the present invention have higher densities and velocities of detonation than the comparable explosives containing PETN, and compositions 5 12 to 15 containing higher explosive loadings are, in addition, markedly superior to the comparable PETN-containing compositions in their ability to withstand exposure to normal and low (subzero) temperature without becoming so brittle as to be inflexible and subject to cracking and crazing. Such higher loadings are preferred for some applications, and the use of PETN at such loadings can lead to processing difficulties, as well as the indicated physical characteristics, as contrasted 10 with RDX and HMX. Furthermore, it should be noted that, in contrast to PETN, which must be used in ultra fine form, RDX and HMX gives products that are adequate for many purposes without any necessity for such fine particle sizes.

WHAT WE CLAIM IS:—

1. A detonating explosive composition comprising a substantially uniform blend consisting essentially of crystalline cyclotrimethylene trinitramine and/or cyclotetramethylenetrinitramine, in amount up to substantially 85%, and of average particle size below 200 microns, high viscosity soluble nitrocellulose in amount substantially $\frac{1}{2}$ to 15%, 25 and a phosphate ester of general formula $O=P-(OR)_3$ and/or a carboxylate ester of general formula



35 wherein the R groups may be the same or different and are C_1 to C_8 functionally-unsubstituted aliphatically-saturated organic radicals, X is a C_1 to C_8 hydrocarbon nucleus, to which the ester groups are attached, m is either 0 or 1 and k is either 2 or 3, in amount substantially 10 to 34.5%, all percentages being by weight.

40 2. A composition according to Claim 1, wherein the amount of nitramine is at least 65% by weight.

45 3. A composition according to Claim 2, wherein the amount of nitramine is at least 68% by weight.

50 4. A composition according to any of Claims 1 to 3, wherein the amount of nitramine is up to 80% by weight.

55 5. A composition according to any of Claims 1 to 4, wherein the average particle size of the nitramine is below 100 microns.

60 6. A composition according to Claim 5, wherein the average particle size of the nitramine is up to 75 microns.

7. A composition according to any of Claims 1 to 6, wherein the average particle size of the nitramine is at least 1 micron.

65 8. A composition according to any of Claims 1 to 7, wherein the amount of nitrocellulose is at least 2% by weight.

9. A composition according to any of Claims 1 to 8, wherein the amount of nitrocellulose is up to 8% by weight.

65 10. A composition according to Claim 9, wherein the amount of nitrocellulose is up to 4% by weight.

70 11. A composition according to any of Claims 1 to 10, wherein the amount of the ester is at least 12% by weight.

75 12. A composition according to any of Claims 1 to 11, wherein the amount of the ester is up to 30% by weight.

80 13. A composition according to any of Claims 1 to 12, wherein the weight of ester is at least four times the weight of the nitrocellulose.

85 14. A composition according to any of Claims 1 to 13, wherein the R groups are hydrocarbyl groups.

85 15. A composition according to any of Claims 1 to 14, wherein the R groups contain up to 4 carbon atoms.

90 16. A composition according to any of Claims 1 to 15, wherein X is a benzenoid nucleus.

90 17. A composition according to any of Claims 1 to 15, wherein X is a saturated nucleus.

95 18. A composition according to Claim 17, wherein X is an acyclic nucleus.

95 19. A composition according to Claim 18, wherein the ester is, at least in part, an alkyl ester of 1,2,3-propane tricarboxylic acid.

100 20. A composition according to Claim 19, wherein the ester is, at least in part, the tributyl ester.

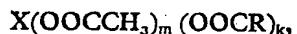
100 21. A composition according to any of Claims 1 to 20, wherein the ester is, at least in part, any of those hereinbefore specifically mentioned.

105 22. A composition according to any of Claims 1 to 21, substantially as hereinbefore described.

105 23. A composition according to Claim 22, substantially as described in any of the foregoing Examples.

110 24. A process for preparing an explosive composition comprising intimately blending together to form a uniform blend crystalline cyclotrimethylenetrinitramine and/or cyclotetramethylenetrinitramine of average particle size below 200 microns in amount up to substantially 85%, high viscosity soluble nitrocellulose in amount substantially $\frac{1}{2}$ to 15%, and a phosphate ester of general formula $O=P-(OR)_3$ and/or a carboxylate ester of general formula

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wherein the R groups may be the same or different and are C_1 to C_8 functionally-unsubstituted aliphatically-saturated organic radicals, X is a C_1 to C_8 hydrocarbon nucleus, to which the ester groups are attached, m is

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either 0 or 1 and k is either 2 or 3, in amount substantially 10 to 34.5%, all percentages being by weight. 45

25. A process according to Claim 24, wherein the components and/or amounts are as claimed in any of Claims 2 to 21.

26. A process according to Claim 24 or 25, wherein at least some of the nitramine is precoated with the ester before blending. 50

10 27. A process according to Claim 26, wherein the amount of ester coating is at least 5% based on the total weight of nitramine and ester coating.

15 28. A process according to Claim 26 or 27, wherein the amount of ester coating is up to 25%, based on the total weight of nitramine and ester coating. 55

20 29. A process according to any of Claims 24 to 28, wherein some of the nitramine is blended with substantially all of the plasticizer before blending with the nitrocellulose.

25 30. A process according to Claim 29, wherein the rest of the nitramine is blended into the composition after substantially all of the nitrocellulose has been blended in. 60

30 31. A process according to Claim 29 or 30, wherein the amount of nitramine blended with the plasticizer before blending with the nitrocellulose is at least 50% by weight of the nitramine. 65

35 32. A process according to Claim 31, wherein the amount of nitramine blended with the plasticizer before blending with the nitrocellulose is at least 65% by weight of the nitramine. 70

35 33. A process according to any of Claims 29 to 32, wherein the amount of nitramine blended with the plasticizer before blending with the nitrocellulose is up to 90% by weight of the nitramine. 75

40 34. A process according to Claim 33, wherein the amount of nitramine blended with the plasticizer before blending with the nitrocellulose is up to 85% by weight of the nitramine.

the plasticizer before blending with the nitrocellulose is up to 85% by weight of the nitramine.

35. A process according to any of Claims 24 to 34, wherein the blending is carried out, at least in part, at an elevated temperature.

36. A process according to Claim 35, wherein the elevated temperature is at least 100°F.

37. A process according to Claim 36, wherein the elevated temperature is at least 120°F.

38. A process according to any of Claims 35 to 37, wherein the elevated temperature is up to 150°F.

39. A process according to Claim 38, wherein the elevated temperature is up to 140°F.

40. A process according to any of Claims 24 to 39, wherein the nitrocellulose is added in a substantially dry form.

41. A process according to any of Claims 24 to 40 substantially as hereinbefore described.

42. A process according to Claim 41 substantially as described in any of the foregoing Examples.

43. An explosive composition prepared according to any of Claims 24 to 42.

44. An explosive composition according to any of Claims 1 to 23 or 43 in the form of a shaped article.

45. A composition according to Claim 44, which is a sheet.

46. A composition according to Claim 44, which is a cord.

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